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The Incorporation of Aluminum Hydroxide in Water Based Muds to Stabilize Marcellus Shale Drilling in West Virginia

Hussain Ali Al-Fadhel
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The Incorporation of Aluminum Hydroxide in Water Based Muds to Stabilize Marcellus Shale Drilling in West Virginia

Hussain Ali Al-Fadhel

**Thesis submitted to the College of Human Resources and Education at West Virginia
University in partial fulfillment of the requirements for the degree of Master of Science in
Petroleum and Natural Gas Engineering**

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2012

**Keywords: Marcellus Shale; Inhibition; Mud additives; Swelling; Aluminum
Hydroxide; Drilling Fluids; Mud; precipitation chemistry; Pore pressure transmission**

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Abstract:

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Hussain Ali Al-Fadhel

The purpose of this study was to develop a safe and environmentally acceptable water base mud that will be able to suppress swelling and promote stability to the West Virginian Marcellus shale. The problem was approached by utilizing aluminum precipitation chemistry in the drilling fluid, unlike the traditional cation-exchange approach.

A dynamic swelling meter was designed by modifying a 0.001 mm resolution dial indicator and using a specially programmed smart cable connecting the dial indicator to a computer. The swelling data was acquired by the smart cable every six seconds. As a result, there were 14,400 measured points for each sample for the 24 hours test duration.

The Marcellus shale samples were prepared by cutting a core sample taken from a depth of 6,025 ft by a rotary saw. The original core sample was cylindrical with a diameter of 2-in. and a height of about 20-in. It was cut into smaller sections with the same diameter and a height ranging from {2.5 to 7} mm. The samples were dried in an oven at 70°C until weight was stabilized. The average moisture content of the samples was approximately 1 %.

Mud samples were prepared with Aquagel (natural clay), Soda Ash (Sodium carbonate), potassium alum (potassium double sulfate of aluminum), and Cellex polymer (Carboxymethyl cellulose sodium). All the substances used in developing the mud are biodegradable and were EC50 (half maximal effective concentration) tested. The base mud design had 10 lb/bbl Aquagel, 2 lb/bbl Cellex, and 5 lb/bbl Soda Ash. Later the potassium alum was added to the base mud with different concentrations (0.5, 1, 0.6, and 0.7) lb/bbl for a sensitivity test.

Each water/mud sample was tested on three shale specimens to confirm the results. Swelling test was first conducted with fresh water, which yielded the highest swelling percentage in an average of 5%. The base mud resulted with approximately 3 % swelling and 1.41 % swelling was observed with the 0.5 lb/bbl potassium alum. Also, 1.24 % swelling with the 0.6 lb/bbl potassium alum, and no swelling with the 0.7 and 1 lb/bbl potassium alum muds. From the sensitivity tests on swelling inhibition performance, the 0.7 lb/bbl potassium alum mud was elected to be the best performer. An additional test on the same mud with three shale samples was carried and results were consistent with the previous one.

Rheological measurements were taken for all the mud samples with American Petroleum Institute (API) standards and were based on the Bingham fluid model. The mud density didn't change with different potassium alum concentrations and remained at 8.8 lb/gal. This was due to the small concentrations incorporated in the base mud. All the test muds showed acceptable rheological properties. The best performer mud with 0.7 lb/bbl potassium alum, had a plastic viscosity of 9 cp, apparent viscosity of 10 cp, and a yield point of 2 lb/100 ft².

Filtration properties experienced small changes as the potassium alum concentration was increased. Overall, all mud samples yielded low filtration rates. The 0.7 lb/bbl potassium alum mud had 6.9 mL filtrate during the 30 minutes test with the filter press and yielded a thin 1.3 mm mud cake (API recommended thickness is < 2 mm).

Finally, a drinking water test kit was used to verify any potential health hazards. The kit was based on the U.S. Environmental Protection Agency (EPA) guideline standards and designed to identify eight common water contaminants: bacteria, lead, pesticide, nitrate, nitrite, pH, hardness, and chlorine. The test was done on the filtrate 0.7 lb/bbl potassium alum mud. The sample passed all tests, except for pH and hardness.

Dedication

To my family who supported and encouraged me through all my life. There are no words that can express my gratefulness and respect.

Acknowledgement

I would like to express my gratitude to my teacher and long time academic advisor Dr. Ilkin Bilgesu for his commitment, guidance, and help throughout this study. Also, I want to thank the chairman of the Petroleum and Natural Gas Engineering department at West Virginia University Professor Sam Ameri for his agreement to be on my committee and his vital insights on the subject. As well, I want to thank the former chairman of Safety and Environmental Management Department at West Virginia University Professor Della-Giustina for his agreement to be a member on my committee and his insights on making the subject of this study as safe and environmentally acceptable as possible.

Table of Contents

Abstract.....	ii
Dedication.....	iv
Acknowledgement.....	v
Problem Statement.....	11
Introduction.....	11
Properties of Shale.....	11
Swelling Mechanism:.....	18
Marcellus Shale.....	20
Location and Geology:.....	20
Marcellus shale in West Virginia:.....	22
Drilling Fluid Design.....	25
Drilling Fluid Functions:.....	25
Drilling Fluid Properties:.....	27
Mud density:.....	27
Mud viscosity:.....	28
Gel Strength:.....	30
pH:.....	30
Filtrate Loss:.....	31
Resistivity:.....	31
Drilling Fluid Types:.....	32
Mud Inhibitors:.....	33
Aluminum Chemistry:.....	34
Previous Work.....	37
Experiment Setup and Preparation.....	39
Core samples.....	39
Mud preparation.....	40
Swelling Meter:.....	41
Results and Dissection:.....	43
Swelling Test Results:.....	43
Water Baseline:.....	43
Base Mud:.....	45
Base Mud + 0.5 lb/bbl Potassium Alum:.....	47

Base Mud + 1 lb/bbl Potassium Alum:	49
Base Mud + 0.6 lb/bbl Potassium Alum:	50
Base Mud + 0.7 lb/bbl Potassium Alum:	52
Base Mud + 0.7 lb/bbl Potassium Alum (R vi tion)	53
Swelling Test Results and Summary:	54
Mud Rheology Results and Discussion:	59
Base Mud:	59
Base Mud + 0.5 lb/bbl Potassium Alum:	60
Base Mud + 0.6 lb/bbl Potassium Alum:	60
Base Mud + 0.7 lb/bbl Potassium Alum:	61
Base Mud + 1 lb/bbl Potassium Alum:	61
Filter Press Test results and Dissection:	63
Resistivity Test Results:.....	66
Filtrate as Drinking water test Results:	69
Conclusion	71
Works Cited	72
Appendix.....	74
Swelling Experiments Log.....	74

List of Figures

FIGURE 1: MUD RING CAUSED BY SHALE (RAMIREZ, ET AL. 2005).....	13
FIGURE 2: WASHOUT ILLUSTRATION.....	14
FIGURE 3: BIT BALLING (WELLS, ET AL. 2008).....	14
FIGURE 4: WATER ABSORPTION BY SHALE FORMATION.....	15
FIGURE 5: FORMATION SWELLING AND CAVING RESULTING IN STUCK PIPE.....	15
FIGURE 6: SWELLING, AND CAVING RESULTING IN HIGH DRAG AND SLOW PENETRATION.....	16
FIGURE 7: WELLBORE SIMULATION ON PIERRE SHALE AT SCHLUMBERGER CAMBRIDGE RESEARCH IN CAMBRIDGE, ENGLAND.....	17
FIGURE 8: SHALE PORE SPACE.....	18
FIGURE 9: SWELLING CLAY PLATELET.....	18
FIGURE 10: PORE-PLATELET UNIT.....	19
FIGURE 11: DIPOLE MOMENT OF WATER MOLECULE.....	19
FIGURE 12: MAP OF MARCELLUS SHALE FORMATION (WEST VIRGINIA GEOLOGICAL AND ECONOMIC SURVEY).....	20
FIGURE 13: GENERALIZED STRATIGRAPHIC NOMENCLATURE FOR THE MIDDLE DEVONIAN STRATA IN THE APPALACHIAN BASIN.....	21
FIGURE 14: MARCELLUS THICKNESS MAP IN FEET (WEST VIRGINIA GEOLOGICAL AND ECONOMIC SURVEY).....	22
FIGURE 15: MARCELLUS PRESSURE REGIMES (WEST VIRGINIA GEOLOGICAL AND ECONOMIC SURVEY).....	23
FIGURE 16: MARCELLUS WELLS AND PERMITS MAP IN WV (NOV.2011) (WEST VIRGINIA GEOLOGICAL AND ECONOMIC SURVEY).....	24
FIGURE 17: MUD BALANCE.....	27
FIGURE 18: VISCOMETER.....	28
FIGURE 19: PH PAPER.....	30
FIGURE 20 :FILTER PRESS.....	31
FIGURE 21: RESISTIVITY METER.....	31
FIGURE 22: MARCELLUS SHALE X-RAY DIFFRACTION (BOYCE AND CARR 2009).....	34
FIGURE 23: CORE SAMPLES IN THE OVEN.....	39
FIGURE 24: SWELLING METERS.....	41
FIGURE 25: DIAL INDICATOR CONTACT POINTS (HTTP://WWW.AUTO-MET.COM).....	42
FIGURE 26: CORE SAMPLE TO BE TESTED.....	42
FIGURE 27: WATER BASELINE SWELLING SUMMARY.....	43
FIGURE 28: DYNAMIC SWELLING MEASRMENTS (WATER BASELINE).....	44
FIGURE 29: DYNAMIC SWELLING MEASRMENTS (WATER BASELINE).....	44
FIGURE 30: BASE MUD SWELLING SUMMARY.....	45
FIGURE 31: DYNAMIC SWELLING MEASRMENTS (BASE MUD).....	46
FIGURE 32: DYNAMIC SWELLING MEASRMENTS (BASE MUD).....	46
FIGURE 33: (0.5 LB/BBL POTASSIUM ALUM) MUD SWELLING SUMMARY.....	47
FIGURE 34: DYNAMIC SWELLING MEASRMENTS (0.5 LB/BBL POTASSIUM ALUM) MUD.....	48
FIGURE 35: DYNAMIC SWELLING MEASRMENTS (0.5 LB/BBL POTASSIUM ALUM) MUD.....	48
FIGURE 36: DYNAMIC SWELLING MEASRMENTS (1 LB/BBL POTASSIUM ALUM)MUD.....	49
FIGURE 37: 0.6 LB/BBL POTASSIUM ALUM MUD SWELLING SUMMARY.....	50
FIGURE 38: DYNAMIC SWELLING MEASRMENTS (0.6 LB/BBL POTASSIUM ALUM) MUD.....	51
FIGURE 39: DYNAMIC SWELLING MEASRMENTS (0.6 LB/BBL POTASSIUM ALUM) MUD.....	51
FIGURE 40: DYNAMIC SWELLING MEASRMENTS (0.7 LB/BBL POTASSIUM ALUM) MUD I.....	52
FIGURE 41: DYNAMIC SWELLING MEASRMENTS (0.7 LB/BBL POTASSIUM ALUM) MUD II.....	53
FIGURE 42: SWELLING RESULTS SUMMARY, ONE HOUR SCALE.....	54

FIGURE 43: COMPARISON OF SWELLING PROFILES I.....	55
FIGURE 44: COMPARISON OF SWELLING PROFILES II	56
FIGURE 45: PLASTIC VISCOSITY AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION	62
FIGURE 46: APPARENT VISCOSITY AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION.....	62
FIGURE 47: BINGHAM YIELD POINT AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION.....	62
FIGURE 48: FILTRATE VOLUME AT DIFFERENT CONCENTRATIONS OF POTASSIUM ALUM	64
FIGURE 49: FILTRATE VOLUME WITH DIFFERENT CONCENTRATIONS OF POTASSIUM ALUM	64
FIGURE 50: MUD CAKE THICKNESS AT DIFFERENT POTASSIUM ALUM CONCENTRATIONS	65
FIGURE 51: MUD CAKE THICKNESS AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION	65
FIGURE 52: FILTRATE RESISTIVITY AT DIFFERENT POTASSIUM ALUM CONCENTRATIONS.....	66
FIGURE 53: FILTRATE RESISTIVITY AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION.....	67
FIGURE 54: MUD CAKE RESISTIVITY AT DIFFERENT POTASSIUM ALUM CONCENTRATIONS	67
FIGURE 55: MUD CAKE RESISTIVITY AS A FUNCTION OF POTASSIUM ALUM CONCENTRATION	68

List of Tables

TABLE 1 SURFACE AREA OF SOME CLAY MINERALS (SABINE 1996).....	12
TABLE 2: MUD COMPONENT PROPERTIES	40
TABLE 3: WATER BASELINE SWELLING RESULTS	43
TABLE 4: BASE MUD COMPOSITION	45
TABLE 5: BASE MUD SWELLING RESULTS	45
TABLE 6: 0.5 LB/BBL POTASSIUM ALUM MUD COMPOSITION.....	47
TABLE 7: (0.5 LB/BBL POTASSIUM ALUM) MUD SWELLING RESULTS	47
TABLE 8: 1 LB/BBL POTASSIUM ALUM MUD COMPOSITION.....	49
TABLE 9: (1 LB/BBL POTASSIUM ALUM) MUD SWELLING RESULTS	49
TABLE 10: 0.6 LB/BBL POTASSIUM ALUM MUD COMPOSITION.....	50
TABLE 11: 0.6 LB/BBL POTASSIUM ALUM MUD SWELLING SUMMARY.....	50
TABLE 12: 0.7 LB/BBL POTASSIUM ALUM MUD COMPOSITION.....	52
TABLE 13: 0.7 LB/BBL POTASSIUM ALUM MUD SWELLING SUMMARY.....	52
TABLE 14: 0.7 LB/BBL POTASSIUM ALUM MUD SWELLING SUMMARY II	53
TABLE 15: BASE MUD RHEOLOGICAL PROPERTIES.....	59
TABLE 16: 0.5 LB/BBL POTASSIUM ALUM MUD RHEOLOGICAL PROPERTIES	60
TABLE 17: 0.6 LB/BBL POTASSIUM ALUM MUD RHEOLOGICAL PROPERTIES	60
TABLE 18: 0.7 LB/BBL POTASSIUM ALUM MUD RHEOLOGICAL PROPERTIES	61
TABLE 19: 1 LB/BBL POTASSIUM ALUM MUD RHEOLOGICAL PROPERTIES	61
TABLE 20: FILTER PRESS TEST RESULTS SUMMARY	63
TABLE 21: FILTRATE & MUD CAKE RESISTIVITIES RESULTS SUMMARY AT DIFFRENT CONCEINTRATION OF POTASSIUM ALUM.....	66
TABLE 22: WATER SAFETY KIT RESULTS SUMMARY	70

Problem Statement

Introduction

One of the major problems in the petroleum industry is wellbore instability. This drilling problem can result in drilling delays, increasing drilling cost, and in sometimes leads to the abandonment of the drilling operation. Drilling operations are very costly by themselves and well instability problems cost the oil industry one billion U.S dollars annually.

Shales account for three fourths of drilled formations. Nonetheless, 90% of the wellbore instability problems are shale related (Patel 2009). Shale rock instability has been well studied, chemically, mechanically, and even on the micro scale. However, shales are still problematic to deal with, not only in the petroleum industry, but in the mining and construction industries as well. There are around nineteen major shale gas plays in the United States alone. The variation of shales depends on many factors, such as mineralogy, age, and depositional environment. All these factors alter the rock chemical and physical characteristics. However, almost all shales share the instability characteristic and they vary in how unstable they are (Halliburton 2008).

Properties of Shale

Shales are defined as laminated clay-bearing sedimentary rocks with less than 0.1 microdarcy. Typically, they have a very finely laminated structure and inter-bedded with sandstone or limestone. Bedding layers thickness can range from a millimeter to hundreds of meters. Moreover, these bedding layers are classified as transversely isotropic. This means that material properties along the bedding planes are different from the plane perpendicular to the bedding (Geology.com)

Most shale rocks bear clay minerals within their matrix. These clay minerals are largely kaolinite, montmorillonite and illite and they are expandable in a process called hydration (Lal 1999). When the clays within shale come in contact with water, hydrogen bonding on the clay surface occurs. Also, a strong bonded molecular layer of water form on the basal crystal surface. More molecular water layers form, with each layer bonding being weaker (D.Clark and Saddok 1993). Moreover, depending on the clay lattice structure, more water is absorbed between layers as well as particle surface. Surface hydration of shale is strong and dependent on the surface area of clays present in their structure. Water is drawn into the shale particle by osmotic force. This force is governed by the salt concentration in the shale and liquid. The hydration of clays results in major problems in drilling and completing oil and gas wells, mainly well instability. Table 1 lists the surface area of clay minerals.

Clay Mineral	Surface Area m^2/g
hematite	10.9
goethite	63.1
amorphous Fe oxide	222.7
Aluminum Oxid C	102.9
gibbsite	56.5
calcite	22
KGa-1 kaolinite	9.14
KGa-2 kaolinite	19.3
SWy-1 montmorillonite	18.6
SAz-1 montmorillonite	48.9
STx-1 montmorillonite	70.3
IMt-1 illite	24.9

Table 1: Surface area of some clay minerals (Sabine 1996)

Shale formations are very challenging to drill and thus expansive too. The main reason behind this is wellbore instability. This drilling problem can cause drilling delays and therefore increasing the drilling cost significantly (Halliburton 2008). Unfortunately, wellbore instability problems can result in the abandonment of a drilling operation. These problems include:

1. Swelling:

Clay swelling occurs when water enters the formation. Clay swelling can be caused by ion exchange or changes in salinity. The nature of the reaction depends on the structure of the clays and their chemical state at the moment of contact.

2. Heaving and sloughing:

This occurs when a partial or complete collapse of the walls of a hole resulting from internal pressures primarily caused by swelling. As a result, the drilling bit and the drill pipe might get stuck with shale cuttings adherence to them. This is known as “mud rings” (Figure 1)

Figure 1: Mud Ring caused by shale (Ramirez, et al. 2005)

3. Washout:

A washout in an openhole section is larger than the original hole size or size of the drill bit. It can be caused by excessive bit jet velocity, soft or unconsolidated formations, in-situ rock stresses, mechanical damage by “Bottom Hole Assembly“ (BHA) components, and swelling or weakening of shale as it contacts water (Figure 2).

Figure 2: Washout Illustration

4. Bit balling:

Bit balling refers to the adhesion of sticky drill cuttings to the drill pipe, drill collar, and between the teeth of the drilling bit. A bit with such material attached to it is called a balled-up bit (Figure 3).

Figure 3: Bit balling (Wells, et al. 2008)

5. Stuck pipe:

Stuck pipe is a drill string that cannot be rotated or moved vertically. There are many reasons that can cause a stuck pipe such as an inadequate hole cleaning, high mud weight, caving and swelling. For example, when shale swells, the diameter of the hole could shrink causing the bit to get stuck, or a caving that will cause the drilling pipe to get stuck at that point (Figures 4 &5).

Figure 4: Water absorption by shale formation

Figure 5: Formation swelling and caving resulting in stuck pipe

6. High drag and slow penetration rate:

All the heaving, balling up, and swelling that aren't enough to get the pipe stuck, will at least cause high drag and the drilling string due to high friction and load. This will slow the penetration speed and even might damage parts of the drilling string because of the high load, in addition to low drilling efficiency (Figure 6).

Figure 6: Swelling, and caving resulting in high drag and slow penetration

It is important to know that some of these problems can be connected to each other. For example, hole sloughing, bit baling, and hole fill can cause high drag and slow penetration. This problem can be caused by formation swelling and/or cuttings swelling in the bore hole. Stuck pipe can occur from swelling and/or hole fill due to the insufficiency of wellbore cleaning due to cutting swelling or shale dispersion. These problems can also cause other problems like, poor logging (D.Clark and Saddok 1993).

Schlumberger Cambridge Research in Cambridge, England, performed a test on Pierre shale in a wellbore simulator. The sample was exposed to mud containing fresh water and bentonite gel. Water has entered the shale causing it to swell and weakening the formation. Continuous flow of mud has eroded the borehole leaving an enlarged hole that would be hard to log and complete.

Figure 7: Wellbore simulation on Pierre shale at Schlumberger Cambridge Research in Cambridge, England (Lal. 1999)

Swelling Mechanism:

The swelling characteristics of any shale are dependent on its composition of swelling clays in its matrix. Figure 8 represents a pore space within a shale sample. The pore space has some water in it with water activity $a_{w,pore}$ and pore pressure P_o . Figure 9 represents a clay platelet space which is negatively charged and has water with water activity $a_{w,platelet}$. The pore space and the clay platelet are connected forming a pore-platelet unit (Figure 10) (Hong Wang 2010). In general water activity in the clay platelet is lower than its counterpart of the pore space. In water, oxygen attracts electrons much more strongly than hydrogen. As a result, net positive charge on the hydrogen atoms, and a net negative charge on the oxygen atom are formed; giving each water molecule a net dipole moment (Figure 11). The highly negatively charged clay platelet attracts water molecules and traps them within in quest of charge equilibrium (Lal. 1999). This causes the clay platelet to expand. Additional swelling takes place by the electrostatic repulsion caused by the water molecules orientation in the clay platelet.

Figure 8: Shale pore space

Figure 9: Swelling clay platelet

Figure 10: pore-platelet unit

Figure 11: dipole moment of water molecule

Marcellus Shale

Location and Geology:

The Marcellus shale is a marine sedimentary rock found in the North East of the United States of America. Marcellus shale extend from the northern tier of New York to northern and western Pennsylvania, eastern Ohio, western Maryland and most of West Virginia. The Marcellus Shale covers an area of approximately 95,000 square miles (Figure 12). The average thickness ranges from 50 ft to 200 feet. Generally, it becomes thicker to the east. It is mostly black in color; however, shallow shale located in the upper portion of the formation is lighter in color. It has been estimated that the entire play holds between 516 to 1,500 Tcf with recent estimated ultimate recovery (EUR) of 489 Tcf. (Projecting the Economic Impact of Marcellus Shale Gas Development in West Virginia 2010)

Figure 12: Map of Marcellus shale formation (West Virginia Geological and Economic Survey)
(<http://www.wvgs.wvnet.edu/>)

The Marcellus is the lowest unit of the Devonian age Hamilton Group, and is divided into several sub-units (Figure 13). It contains interbedded limestone layers due to sea level variation during its deposition around 400 million years ago. The black shale was deposited in relatively deep water deprived of oxygen.

Sys-tem	Ohio	N. Virginia and West Virginia	Western Maryland	Western Pennsylvania	Northwestern New York
Middle Devonian		Harrell Shale	Harrell Shale	Harrell Shale	Genesee Fm.
	Olentangy Shale	Tully Limestone		Tully Limestone	Tully Limestone
		Mahantango Formation	Mahantango Formation	Mahantango Formation	Moscow Shale
					Ludlowville Shale
	Prout Limestone				Skaneateles Shale
	Plum Brook Shale				
	Delaware Limestone	Marcellus Shale	Marcellus Shale	Marcellus Shale	Marcellus Shale
Lower Dev.	Columbus Limestone	Huntersville Chert	Needmore Shale	Selinsgrove Limestone	Onondaga Limestone
	Bois Blanc Limestone			Needmore Shale	Bois Blanc Fm.

Figure 13: Generalized stratigraphic nomenclature for the Middle Devonian strata in the Appalachian Basin

(Boyce and Carr 2009)

Marcellus shale in West Virginia:

In West Virginia Marcellus Shale is present throughout the state, except in the very southwest and some of the very east parts of the state. It covers an estimated area of 6,985,000 acres of West Virginia, with an estimated EUR within the range of 98 Tcf to 150 Tcf (Projecting the Economic Impact of Marcellus Shale Gas Development in West Virginia 2010). The thickness varies across the state, where it's thickest in northeast-central counties and gradually gets thinner towards the southwest (Figure 14).

Figure 14: Marcellus Thickness Map in feet (West Virginia Geological and Economic Survey)
(<http://www.wvgs.wvnet.edu/>)

The Marcellus shale is associated with different pressure regimes in West Virginia. In general it is under-pressured in the southwest, normal to potentially over-pressured in the northeast, and a transitional regime in the area in between (Figure 15).

Figure 15: Marcellus Pressure Regimes (West Virginia Geological and Economic Survey)

(<http://www.wvgs.wvnet.edu/>)

As of November 10, 2011 according to West Virginia Geological and Economic Survey (WVGES), 1515 wells have been completed in the Marcellus shale formation and 1352 new drilling permits have been issued. Figure 16 the map of Marcellus shale wells (red) and permits (yellow) in West Virginia.

Figure 16: Marcellus Wells and permits map in WV (Nov.2011) (West Virginia Geological and Economic Survey)

(<http://www.wvgs.wvnet.edu/>)

- Completed Wells with a Marcellus Pay Zone
- Permitted Wells with Marcellus/Devonian and deeper

Drilling Fluid Design

Drilling Fluid Functions:

Remove cuttings from well

As the drilling fluid is pumped through the drill string, it carries the rock excavated by the drill bit up to the surface. The efficiency of this process depends on the cuttings size, shape, density, and mud circulation velocity.

Suspend and release cuttings

In static conditions, the mud should be able to suspend cuttings and preventing them from settling at the bottom of the hole. The settling of cutting at the bottom is called sagging and can cause many problems, such as stuck pipe and bridging.

Control formation pressures

In over-balanced conditions, the pressure exerted by the mud is higher than the formation pressure. This allows well control, which means that uncontrollable flow of formation fluids into the wellbore and preventing blow-outs. The hydrostatic pressure of the drilling fluid is manipulated by its density, where:

$$\text{Hydrostatic Pressure}(\text{psi}) = \rho \times 0.052 \times D$$

Where:

ρ is the mud density in lb/gal

0.052 is a unit conversion factor

D is the true vertical depth in ft

Seal permeable formations

As the mud column pressure exceeds formation pressure, mud filtrate invades the formation, and solids from the mud accumulate on the walls of the hole forming what's known as mud cake. A good mud cake should be thin and have low permeability. This will protect the formation from damage and provide support to the borehole wall.

Maintain wellbore stability

In low permeability formations such as shale, mud cake can't be formed. This leaves the formation vulnerable to swelling and erosions. However, the chemical composition of the drilling fluid should provide means of wellbore stability. Usually, this is achieved by using adding swelling inhibitors, such as salts.

Cool and lubricate the bit.

Heat is generated at the drill bit from friction against rock and in drill pipe rubbing against well casing. As the mud is circulated through the drill string, it transfers the heat away. This process protects the drilling string from getting damaged, providing it with a longer operating-life.

Drilling Fluid Properties:

Mud density:

Mud weight is measured in lb/gal (pounds per gallon) with a mud balance. In a water based mud, the weight is increased by adding solids such as clay or weighing agents. As mentioned earlier, the mud column exerts hydrostatic pressure against the wellbore wall. Therefore, depending on the formation pressure being drilled, mud density is manipulated to overcome that pressure. High pressure formations require higher mud densities to prevent blow-outs from occurring. Nonetheless, nominal and low formation pressure will require lighter muds; just enough to over-balance the pressure. Too high mud weight will cause formation damage and might cause unwanted formation fractures. Given the formation pressure trends and depths of the Marcellus shale in West Virginia, drilling fluids ranging from water with 8.32 lb/gal to 10 lb/gal will most likely be suitable.

Figure 17: Mud Balance

Mud viscosity:

Plastic Viscosity (PV):

It is parameter based on the Bingham plastic model derived from the slope of the shear stress-shear rate. Using a Viscometer, PV can be derived from the 600 rpm reading minus the 300 rpm reading and it is measured in centipoises (cp). In general, low plastic viscosity muds yield faster penetration rates due to the low viscosity of mud at the bit. High plastic viscosity muds have higher capabilities in lifting cuttings from the bottom of the borehole to the surface. However, high plastic viscosity muds can cause higher frictional pressure losses which will affect the drilling efficiency.

$$PV = 600_{rpm \text{ reading}} - 300_{rpm \text{ reading}}$$

Figure 18: Viscometer

Apparent Viscosity:

Apparent viscosity is the viscosity of a fluid measured at the shear rate specified by API. In the Bingham plastic rheological model, apparent viscosity is one-half of the viscometer dial reading at 600 rpm.

$$PA = \frac{1}{2} 600_{rpm \text{ reading}}$$

Yield Point (YP):

Yield point is defined as the resistance of the drilling fluid to initial flow, or the stress needed to start fluid movement. YP is derived using the viscometer by subtracting plastic viscosity PV from the 300 rpm dial reading on the viscometer and it is measured in $\text{lbf}/100\text{ft}^2$. This value is used to determine the ability of the drilling fluid to carry cuttings to the surface. Non-Newtonian fluids are associated high yield points, and are able to carry cuttings better than lower yield point fluids of the same density. Deflocculants are used to increase the yield point of a drilling fluid, while Flocculants are used to decrease it.

$$YP = PV - 300_{rpm \text{ reading}}$$

Gel Strength:

It is the ability of the drilling fluid to suspend cuttings in static condition. Gel strength is derived from the viscometer as well. The drilling fluid has to be agitated by setting the viscometer to 600 rpm. Then the viscometer is set to zero rpm and the mud is left to settle for 10 seconds. The viscometer then is set to 3 rpm and the maximum dial reading is reported in lbf/100 ft². Also, there is a 10 minutes gel strength test that can be performed in the same manner.

pH:

It is a measure of the acidity or basicity of an aqueous solution. The pH scale ranges from 0 to 14, where solutions with a pH less than 7 are acidic and solutions with a pH greater than 7 are basic or alkaline. pH can be measured using pH paper or digital pH meter.

Figure 19: pH Paper

Filtrate Loss:

This test shows the fluid loss behavior of the drilling fluid as it is compressed with 100 psi pressure in a cylinder against a filter paper for 30 minutes. As the fluid is filtered out, solids from the mud will accumulate on the filter paper forming a mud-cake. A good drilling fluid will form a thin and resilient mud-cake with low permeability. This prevents the drilling fluid from invading permeable formations and providing support to the wellbore wall.

Figure 20 :Filter Press

Resistivity:

The resistivity of any drilling fluid depends on its composition, and it is an important parameter in resistivity logging. In resistivity logging high resistivity could potentially be a hydrocarbon bearing zone. The mud resistivity is measured using a resistivity meter and reported in ohms.

Figure 21: Resistivity Meter

Drilling Fluid Types:

There are three main types of drilling fluids; water-based (WBM), oil-based (OBM) and synthetic-based (SBM). Technically, OBM and SBM drilling fluids are the best suited for drilling shale formations because they don't interact with shale (Ramirez, et al. 2005). They provide stability of borehole and cuttings, and efficient solids removal. Oil and synthetic oil based muds have high lubricity, higher penetration rate and withstand higher temperatures than water-based muds.

Regardless of their high performance, they have a downside. They are very expensive, difficult to mix, prone to lost circulation, less environmentally acceptable than water-based muds. Furthermore, the application of OBM or SBM requires the following:

1. Special authorization of usage in the area.
2. Expensive cuttings disposal protocols.
3. Long-term legal liability.

The disadvantages of OBM and SBM drilling fluids have led to a much safer and cheaper approach. HPWBMs (high performance water-based muds) have been developed over time. By adding shale inhibitors, stabilizers and lubricants, the gap in performance between OBM and WBM drilling fluids became smaller. These additives can decrease or retard shale from interacting with the water in the drilling fluid and provide wellbore stability (Arvind, et al. 2007).

Mud Inhibitors:

Mud additives are commonly considered as drilling mud contaminants. Salt and calcium sulfate are called inhibitors when purposely added to drilling muds in order to prevent or retard the hydration of clay in shale formations. There are many additives, traditionally salts such as potassium chloride (KCl) that can be used to decrease water activity in the mud. The salt in the drilling fluid dissolves into ions; anions which are negatively charged and cations which are positively charged. The negatively charged clay platelets in the shale attract the positively charged cation in the mud in a process called “cation exchange.” After this process takes place, the clay platelets reach electrical charge equilibrium eliminating the need to attract the much larger and less charged water molecules to reach such state.

The challenge remains of what additives to use and in what concentration. This is because no two shales are alike. Different shales have different clay minerals with different concentrations and different matrix structures. Thus, a mud that is suited for Barnett shale might not be suitable for Marcellus shale and vice versa.

Aluminum Chemistry:

The Marcellus shale has high concentration of illite clay, which is prone to dispersion. The illite makes up anywhere from 20% to 80%, making Marcellus shale very fragile and dispersive. Also, the presence of calcite and kaolinite makes the Marcellus prone to swelling. Figure 22 shows the mineral composition of a Marcellus shale sample from Pennsylvania. The mineralogy of the sample was obtained using X-ray Diffraction technique (XRD). It shows that illite makes up for 24.6 %, 0.3% calcite, and 2.6% kaolinite of the shale. Though this was a sample from Pennsylvania, it should give a good indication of the Marcellus in West Virginia due to close geographical proximity of the two states.

Figure 22: Marcellus Shale X-ray Diffraction (Boyce and Carr 2009)

Traditional mud inhibitors that depend only on cation exchange, can minimize swelling, but can't prevent borehole stability problems. The cation exchange approach gives the charge equilibrium for the clay and preventing it from swelling, but leaves the pores open. This leaves the shale pores prone to the hydrostatic pressure from the mud column. As a result, pore pressure around the wellbore will be elevated with nowhere to dissipate this pressure due to low

permeability of the shale. This stress will fracture and break the shale front. Pore pressure transmission problem becomes more serious in high illite shale such as the Marcellus.

The application of aluminum salts as shale stabilizers in drilling fluids was first described in 1973. However, the mechanism of their ability to do so wasn't fully understood at time. The development of pore pressure transmission in shale has lead to the identification of aluminum salts shale inhabitation mechanisms.

The aluminum chemistry approach to shale stability is based on altering the shale matrix, in contrast to the widely used ion exchange approach. When aluminum salt is added to drilling mud systems, it produces aluminate, $Al(OH)_4^-$. The aluminate will remain soluble as long as the pH is maintained at 10 or higher. As filtrate from the mud encounters near wellbore pore space fluids at lower pH, the soluble aluminate starts to precipitate as aluminum hydroxide, $Al(OH)_3$. Aluminum hydroxide precipitation in the pore spaces at the surface of the shale will create an inner mud cake. This inner mud cake will significantly reduce pore pressure transmission and provide extra support to the shale.

In this study, potassium alum was used as a source of aluminum hydroxide. Potassium alum is the potassium double sulfate of aluminum and its chemical formula is $KAl(SO_4)_2$. It is commonly found in its dodecahydrate form as $KAl(SO_4)_2 \cdot 12(H_2O)$. It occurs naturally as a sulfate mineral, usually on rocks in areas of weathering and oxidation of sulfide minerals and potassium bearing minerals. It is commonly used in water purification, body deodorants, after shaving products, stop-bleeding and cosmetic products and in medicine.

This aluminum was chosen based on three main factors:

- A. Safe to handle and environmentally acceptable.
- B. Available in commercial quantities with acceptable prices (\$31.50/ 50 lb sack)
(Gallade Chemical 2012).
- C. Unlike other aluminum salts, it produces potassium ion; instead of adding potassium chloride KCl to the mud later to increase its inhabitation performance and eliminating the chlorine byproduct as well.

Also Soda Ash, Sodium carbonate Na_2CO_3 , was used in the mud as a pH regulator to maintain stable alkaline conditions for the aluminate to remain soluble in the mud. It is commonly added in pools to neutralize the acidic effects of chlorine and raise pH.

Previous Work

Few published work related to aluminum/precipitating chemistry in drilling fluids, since pore pressure transmission measuring techniques have been only developed recently (Stowe, et al. 2001) . Nonetheless, the aluminum chemistry in drilling fluids has been applied in Louisiana USA, Gulf of Mexico, South America, Norway, and Algeria (Halliday, et al. 1993) (Ramirez, et al. 2005).

M. Ramirez, D. Clapper, P. Kenny published their work on the same subject in 2005. Their work was focused on developing a water based mud to be used in exploration wells in a very environmental sensitive area in the Upper Magdalena Valley of Colombia. The targeted formation was the Villeta shale, which was associated with major well stability and stuck-pipe issues. Shale cuttings from a nearby well were taken to perform test on them with the aluminum inhibitor mud.

Hydration and pore-pressure transmission tests were performed on the samples with different drilling fluids. Their studies showed that the use of aluminum complex in water resulted in 12% hydration, whereas it was 50% in fresh water. The mud was put to use in several wells and no bit balling or stability problems were encountered and the mud was deemed successful. (M., D. and G., et al. 2005)

The same group of researchers also published their second case study in 2006. This study was conducted on the Napo shale of Yuralpa Field, Ecuador. Wellbore stability was the major drilling problem in this field. Moreover, there were environmental concerns of using oil based mud. Their work lead to the use of water based mud (Barite) incorporated with Aluminum salt as an inhibitor and a stabilizer. (Ramirez, et al. 2006)

They conducted dispersion tests on the Napo shale, which showed 58.2% dispersion in fresh water, 38.2% dispersion in xanthan gum mud and 14.2% in the aluminum inhibitor mud. Their mud design yielded great results as the stability problems were eliminated and achieved higher penetration rates compared to traditional KCl muds.

In both published case studies, the name of the aluminum salt used was not disclosed. Both publications referred to it as AHC (Aluminum Hydroxide Complex) source. However, they stated the concentration of it in their design. The concentration of aluminum hydroxide complex source used was ranging from 1 lb/bbl in the Villeta shale and 3.5 for the Napo shale. Also, they added 5 lb/bbl KCl in their mud design to further enhance the inhibition performance of the drilling fluid.

M. Ramirez and his team of researchers have enhanced the understanding of aluminum chemistry in drilling fluids and showed its applicability in the field. In this study their general concept is incorporated in developing an environmentally acceptable water based mud for the Marcellus shale.

Experiment Setup and Preparation

Core samples

The core samples used in this study were obtained from a well drilled in West Virginia. The core was taken from a depth of 6025 ft and it was well preserved in a tight plastic enclosure. The original core sample was cylindrical with a diameter of 2-in. and a height of 20-in. It was cut into smaller sections with the same diameter and a height ranging from {2.5 to 7} mm. The samples were dried in an oven at 70°C until weight was stabilized. The average moisture content of the samples was approximately 1 %. A total of 39 samples have been prepared. Figure 23 shows some of the samples in the core oven during the drying process.

Figure 23: Core samples in the oven

Mud preparation

To maintain consistency, the base mud was chosen to be 10 lb/bbl (pound per barrel) Aquagel clay, 2 lb/bbl Cellex polymer for filtration control, and 5 lb/bbl Soda Ash for pH control. The Potassium Alum was then added in different concentration to be tested with the shale samples. The fluids tested were:

- A. Water: To provide a swelling baseline measure.
- B. Base mud: To provide a reference point to compare the effect of the aluminum additive
- C. Base mud + 0.5 lb/bbl Potassium Alum
- D. Base mud + 0.75 lb/bbl Potassium Alum
- E. Base mud + 1 lb/bbl Potassium Alum

The mud component properties are listed in Table 2:

Component	Chemical Name	Chemical Formula	Application	Specific Gravity @ 20 C	pH	Recommended Concentration
Aquagel	wyoming sodium bentonite	variable	Visocifier	2.65	8--10 (1% Solution)	0-25 lb/bbl
Cellex	Carboxymethyl cellulose sodium	variable	Filtration Control	1.6		0.2-3.5 lb/bbl
Soda Ash	Sodium Carbonate	Na ₂ CO ₃	pH Regulator	2.5	11.3 (1% Solution)	N/A
Potssuim Alum	potassium double sulfate of aluminium	KAl(SO ₄) ₂	Mineral Salt	1.757	3.0-3.5 (10% aq. solution)	N/A

Table 2: Mud component Properties

Swelling Meter:

To measure the swelling, three dial indicators with a 0.001 mm resolution were used. One of the three dial indicators was used as a dynamic swelling meter. The logic series indicators have a data output port. A smart cable with a customized module was used for data acquisition. The smart cable was programmed to acquire data from the dial indicator every six seconds. From one end it connects to the dial indicator via a RS-232 port and from the other to a computer via a normal USB port. The cable automatically inputs the data in a column in Microsoft® Excel. Therefore, the swelling versus time can be plotted to show the swelling profile of the samples.

Figure 24: Swelling Meters

Also, the contact point on each dial indicator was replaced with a wider flat one (Figure 25). This is to minimize the impact of the pen's weight by distributing it on a larger area. Though the weight is small, the swelling measurements are very sensitive.

Figure 25: Dial Indicator contact points (<http://www.auto-met.com>)

Before testing the samples, their initial thickness is measured. To minimize measurement errors caused by surface irregularities of the shale samples, a point and arrow are marked on them (Figure 26). The sample is placed such that the arrow points towards the indicator's holding bar, while the marked point is where the initial thickness and swelling are measured. Also, for each swelling test, a fresh sample was used as they can't be reused due to the alteration of the shale matrix by the aluminum hydroxide precipitation.

Figure 26: Core sample to be tested

Results and Discussion:

Swelling Test Results:

Water Baseline:

This test was performed on samples 17, 19 and 21. The swelling test results were 4.88 %, 5.07%, and 4.93% correspondingly. The average swelling was approximately 5%, which is consistent with the little amount of swelling clay in the Marcellus shale showed by the X-ray Diffraction (Figure 22). The water baseline swelling results are shown in Table 3 and Figure 28.

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
17	3.302	0.161	4.88
19	3.074	0.156	5.07
21	3.427	0.169	4.93
Average			4.96

Table 3: Water Baseline Swelling Results

Figure 27: Water Baseline Swelling Summary

Figure 28: Dynamic Swelling Measurements (Water Baseline)

Figure 29: Dynamic Swelling Measurements (Water Baseline)

From Figures 28 and 29, swelling occurs instantaneously. Primary swelling is fast and stabilizes at around 2.5 % within the first hour. Secondary swelling starts after 16 hours and violently jumps from 2.5 % to around 5 % in less than two hours.

Base Mud:

This test was performed on samples 18, 12 and 14 with base mud. The components of the mud and their concentrations in lb/bbl are shown in table 4. The average swelling was almost 3 %.

The Cellex polymer and Soda Ash had helped in reducing swelling by approximately 2%. Table 5 and Figure 30 show the base mud swelling results.

Component	Concentration
	lb/bbl
Aquagel	10
Cellex	2
Soda Ash	5

Table 4: Base Mud Composition

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
18	4.084	0.120	2.94
12	3.607	0.109	3.02
14	4.162	0.115	2.76
Average			2.91

Table 5: Base Mud Swelling Results

Figure 30: Base Mud Swelling Summary

Figure 31: Dynamic Swelling Measurements (Base Mud)

Figure 32: Dynamic Swelling Measurements (Base Mud)

Figures 31 and 32 show that primary swelling occurred instantaneously to reach 1%. However, the swelling was slower and less aggressive than with water only. Additionally, Secondary swelling occurred after five hours only. The secondary swelling was slower and took around 15 hours to finally stabilize; increasing from 1% to 2.76%.

Base Mud + 0.5 lb/bbl Potassium Alum:

This test was performed on samples 15, 13 and 16. Table 6 shows the mud composition and concentrations in lb/bbl. This test mud was able to lower the swelling to an average of $\approx 1.41\%$ (Table 7 and Figure 33). The Marcellus shale showed high sensitivity to the potassium alum in the mud. The 0.5 lb/bbl potassium alum concentration in the mud was able to reduce the swelling by 1.59 % from base mud which had an average of 3 %.

Component	Concentration
	lb/bbl
Aquagel	10
Cellex	2
Soda Ash	5
Potassium Alum	0.5

Table 6: 0.5 lb/bbl Potassium Alum Mud Composition

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
15	5.574	0.079	1.42
13	6.99	0.098	1.40
16	6.652	0.093	1.40
Average			1.41

Table 7: (0.5 lb/bbl Potassium Alum) Mud Swelling Results

Figure 33: (0.5 lb/bbl Potassium Alum) Mud Swelling Summary

Figure 34: Dynamic Swelling Measurements (0.5 lb/bbl Potassium Alum) Mud

Figure 35: Dynamic Swelling Measurements (0.5 lb/bbl Potassium Alum) Mud

The dynamic swelling meter result showed that the 0.5 lb/bbl potassium alum mud was able to hold the sample from swelling for 3 hours (Figures 34 and 35). The swelling completely stabilized after 14 hours. The whole swelling process with this mud was only about 11 hours. The swelling process was shorter than its counterpart with water or base mud.

Base Mud + 1 lb/bbl Potassium Alum:

This test was performed on samples number 11, 23 and 28. Table 8 shows the mud composition and concentrations in lb/bbl. The 1 lb/bbl potassium alum mud was able to completely suppress swelling (table 8). Also, the dynamic swelling test showed that there was no fluctuation during the whole 24 hours test period (Figure 36). From these results, it was concluded that the potassium alum concentration would be more than 5 lb/bbl and less or equal to 1 lb/bbl. More tests were performed between these two concentrations to make sure that no more that required potassium alum is used in the mud. Table 9 shows the swelling test results for each sample.

Component	Concentration
	lb/bbl
Aquagel	10
Cellex	2
Soda Ash	5
Potassium Alum	1

Table 8: 1 lb/bbl Potassium Alum Mud Composition

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
11	7.128	0	0.00
23	5.66	0	0.00
28	6.107	0	0.00
Average			0.00

Table 9: (1 lb/bbl Potassium Alum) Mud Swelling Results

Figure 36: Dynamic Swelling Measurements (1 lb/bbl Potassium Alum) Mud

Base Mud + 0.6 lb/bbl Potassium Alum:

Samples 3, 5 and 8 were tested with the 0.6 lb/bbl potassium alum mud. In this test, the average swelling was 1.24 % (Table 10 and Figure 37). This mud was able to slightly reduce the swelling of the samples by an average of 0.17 % compared to the 0.5 lb/bbl potassium alum mud. Nonetheless, this result was expected as the concentration of potassium alum was only 0.1 lb/bbl more.

Component	Concentration
	lb/bbl
Aquagel	10
Cellex	2
Soda Ash	5
Potassium Alum	0.6

Table 10: 0.6 lb/bbl Potassium Alum Mud Composition

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
3	5.619	0.070	1.25
5	5.19	0.064	1.23
8	5.764	0.072	1.25
Average			1.24

Table 11: 0.6 lb/bbl Potassium Alum Mud Swelling Summary

Figure 37: 0.6 lb/bbl Potassium Alum Mud Swelling Summary

Figure 38: Dynamic Swelling Measurements (0.6 lb/bbl Potassium Alum) Mud

Figure 39: Dynamic Swelling Measurements (0.6 lb/bbl Potassium Alum) Mud

Figures 38 and 39 from the dynamic measurements showed a very different swelling profile than the 0.5 lb/bbl alum mud. The 0.6 lb/bbl alum mud was able to hold the swelling for only less than 10 minutes, whereas the 0.5 lb/bbl mud was able to do so for 3 hours. Also, the swelling process took a longer time to stabilize. The sample took 18 hours to stabilize and it was increasing almost linearly. The sensitivity of Marcellus shale was observed as the swelling profile completely changed with a small concentration increase of potassium alum.

Base Mud + 0.7 lb/bbl Potassium Alum:

The 0.7 lb/bbl alum mud test was performed on samples number 2, 6 and 10 (Table 13). Table 12 shows the mud composition and concentrations in lb/bbl. Unexpectedly, the mud completely stopped the swelling in all samples. This was a drastic change from the earlier 0.6 lb/bbl alum mud, where it went down from an average of 1.24 % to 0%. This result provided that the 1 lb/bbl potassium alum concentration in the mud was actually more than what it was needed to stop the swelling. The result was very surprising and it was decided to redo the test to re-confirm it. Figure 40 shows the dynamic swelling measurements performed on sample number 10, where it shows no fluctuations occurred during the test.

Component	Concentration
	lb/bbl
Aquagel	10
Cellex	2
Soda Ash	5
Potassium Alum	0.7

Table 12: 0.7 lb/bbl Potassium Alum Mud Composition

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
2	4.815	0	0.00
6	4.307	0	0.00
10	5.024	0	0.00
Average			0.00

Table 13: 0.7 lb/bbl Potassium Alum Mud Swelling Summary

Figure 40: Dynamic Swelling Measurements (0.7 lb/bbl Potassium Alum) Mud I

Base Mud + 0.7 lb/bbl Potassium Alum (Re-Confirmation)

The performance re-confirmation of the 0.7 lb/bbl potassium alum mud was performed on samples number 5, 1 and 7. The results shown in Table 14 were consistent with the earlier test, only one sample varied with a 0.001 mm (0.03 %) making the average swelling of the whole test to be 0.01%. As a result, it was decided that the 0.7 lb/bbl alum mud was the best performer and there was no need to increase the alum concentration. Figure 41 shows the dynamic swelling measurements in seconds.

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
5	4.930	0	0.00
1	3.705	0.001	0.03
7	7.005	0	0.00
Average			0.01

Table 14: 0.7 lb/bbl Potassium Alum Mud Swelling Summary II

Figure 41: Dynamic Swelling Measurements (0.7 lb/bbl Potassium Alum) Mud II

Swelling Test Results and Summary:

Baseline (Water)		Base Mud		0.5 lb/bbl Potassium Alum		0.6 lb/bbl Potassium Alum	
Time h	Swelling %	Time hr	Swelling %	Time hr	Swelling %	Time hr	Swelling %
0	0.00	0	0.00	0	0.00	0	0.00
1	2.25	1	0.86	1	0.00	1	0.26
2	2.25	2	0.86	2	0.00	2	0.66
3	2.25	3	0.86	3	0.00	3	0.68
4	2.25	4	0.86	4	0.32	4	0.69
5	2.25	5	0.86	5	0.32	5	0.75
6	2.25	6	1.11	6	0.32	6	0.78
7	2.25	7	1.27	7	0.78	7	0.83
8	2.25	8	1.27	8	0.78	8	0.87
9	2.25	9	1.37	9	1.04	9	0.94
10	2.57	10	1.37	10	1.04	10	0.95
11	2.57	11	1.56	11	1.04	11	1.02
12	2.57	12	1.56	12	1.04	12	1.04
13	2.57	13	1.71	13	1.04	13	1.09
14	2.57	14	1.92	14	1.40	14	1.09
15	2.57	15	1.92	15	1.40	15	1.16
16	2.57	16	2.07	16	1.40	16	1.16
17	3.47	17	2.38	17	1.40	17	1.20
18	4.73	18	2.38	18	1.40	18	1.23
19	4.73	19	2.64	19	1.40	19	1.25
20	4.73	20	2.69	20	1.40	20	1.25
21	4.73	21	2.75	21	1.40	21	1.25
22	4.93	22	2.75	22	1.40	22	1.25
23	4.93	23	2.75	23	1.40	23	1.25
24	4.93	24	2.75	24	1.40	24	1.25

1 lb/bbl Potassium Alum		0.7 lb/bbl Potassium Alum I		0.7 lb/bbl Potassium Alum II	
Time hr	Swelling %	Time hr	Swelling %	Time hr	Swelling %
0	0.00	0	0.00	0	0.00
1	0.00	1	0.00	1	0.00
2	0.00	2	0.00	2	0.00
3	0.00	3	0.00	3	0.00
4	0.00	4	0.00	4	0.00
5	0.00	5	0.00	5	0.00
6	0.00	6	0.00	6	0.00
7	0.00	7	0.00	7	0.00
8	0.00	8	0.00	8	0.00
9	0.00	9	0.00	9	0.00
10	0.00	10	0.00	10	0.00
11	0.00	11	0.00	11	0.00
12	0.00	12	0.00	12	0.00
13	0.00	13	0.00	13	0.00
14	0.00	14	0.00	14	0.00
15	0.00	15	0.00	15	0.00
16	0.00	16	0.00	16	0.00
17	0.00	17	0.00	17	0.00
18	0.00	18	0.00	18	0.00
19	0.00	19	0.00	19	0.00
20	0.00	20	0.00	20	0.00
21	0.00	21	0.00	21	0.00
22	0.00	22	0.00	22	0.00
23	0.00	23	0.00	23	0.00
24	0.00	24	0.00	24	0.00

Figure 42: Swelling Results Summary, One hour scale

Figure 43: Comparison of Swelling Profiles I

Figure 44: Comparison of Swelling Profiles II

Figures 43 and 44 show the different swelling profiles of the Marcellus shale in the different muds. The water baseline had the highest and fastest swelling. In the fresh water case, primary swelling occurred immediately and reached 2.5 % in the first hour of the test. The swelling was fairly stabilized for the next 15 hours. The secondary swelling increased to around 5 % in less than two hours.

The base mud test showed a better performance, where it slowed the swelling process and lowered it by slightly over 2 %. This was due to the Cellex polymer which had inhabitation qualities. The primary swelling occurred immediately reaching 1% in the first hour and stabilized for 4 hours. Secondary swelling started after that and lasted for 16 hours; increasing swelling from 1% to 2.76%.

The mud with the 0.5 lb/bbl potassium alum dramatically lowered the swelling. Even though the concentration of the inhibitor was small, it was able to lower the swelling to 1.40 % in average compared to base mud. Interestingly, primary swelling started after 3 hours to reach around 0.4 % and stabilized for two hours. Secondary swelling started after 6 hours from the start and continued for 8 hours and increased swelling by 1 % only.

In the case of the 0.6 lb/bbl potassium alum, the swelling was lowered by 0.17 % in average compared to the 0.5 lb/bbl potassium alum mud. However, the swelling profile was altered. Primary swelling started minutes after the test was started to reach approximately 0.8 %. Primary swelling duration was stretched to 18 hours before stabilizing. In this test there was no evidence of secondary swelling, and it seemed that there was only one stretched swelling stage.

The 0.7 lb/bbl and 1 lb/bbl potassium alum muds completely stopped the shale from swelling. Nevertheless, it has been decided the 0.7 lb/ bbl potassium alum mud to be the best performer after testing it on six samples with consistent results.

Mud Rheology Results and Discussion:

Mud Rheology tests were performed on all mud samples. Overall, there was minor rheological alteration from base mud as the potassium alum was added. The mud density remained constant at 8.8 lb/gal. For the base mud, 0.5 lb/bbl and 0.6 lb/bbl potassium alum muds the rheological properties were the same. For these samples, plastic viscosity was 8 cp, 10 cp for apparent viscosity, and 4 lb/100 ft² for yield point. On the other hand, rheological properties started to show small changes at 0.7 lb/bbl and 1 lb/bbl potassium alum mud samples. The 0.7 lb/bbl potassium alum mud had a plastic viscosity of 9 cp, apparent viscosity of 10 cp and yield point of 2 lb/100 ft². The 1 lb/bbl potassium alum mud had a plastic viscosity of 9 cp, apparent viscosity of 9.5 cp and yield point of 1 lb/100 ft². The rheology test results for the (0.5, 0.6, 0.7 and 1) lb/bbl muds are shown in Tables 15, 16, 17, 18 and 19 respectively.

Base Mud:

Density	8.8	lb/gal
---------	-----	--------

ϕ_{600}	ϕ_{300}	ϕ_3 Gel
cp	cp	cp
20	12	5

μ_p	8	cp
μ_a	10	cp
Y_b	4	lb/100 ft ²

Table 15: Base mud rheological properties

Base Mud + 0.5 lb/bbl Potassium Alum:

Density	8.8	lb/gal
----------------	------------	---------------

ϕ_{600}	ϕ_{300}	ϕ_3 Gel
cp	cp	cp
20	12	5

μ_p	8	cp
μ_a	10	cp
Y_b	4	lb/100 ft ²

Table 16: 0.5 lb/bbl potassium alum mud rheological properties

Base Mud + 0.6 lb/bbl Potassium Alum:

Density	8.8	lb/gal
----------------	------------	---------------

ϕ_{600}	ϕ_{300}	ϕ_3 Gel
cp	cp	cp
20	12	4

μ_p	8	cp
μ_a	10	cp
Y_b	4	lb/100 ft ²

Table 17: 0.6 lb/bbl potassium alum mud rheological properties

Base Mud + 0.7 lb/bbl Potassium Alum:

Density	8.8	lb/gal
----------------	------------	---------------

ϕ_{600}	ϕ_{300}	$\phi_{3 \text{ Gel}}$
cp	cp	cp
20	11	4

μ_p	9	cp
μ_a	10	cp
Y_b	2	lb/100 ft ²

Table 18: 0.7 lb/bbl potassium alum mud rheological properties

Base Mud + 1 lb/bbl Potassium Alum:

Density	8.8	lb/gal
----------------	------------	---------------

ϕ_{600}	ϕ_{300}	$\phi_{3 \text{ Gel}}$
cp	cp	cp
19	10	3

μ_p	9	cp
μ_a	9.5	cp
Y_b	1	lb/100 ft ²

Table 19: 1 lb/bbl potassium alum mud rheological properties

Figures 45, 46 and 47 show the change in different rheological properties with respect to Potassium Alum concentration in lb/bbl.

Figure 45: Plastic viscosity as a function of potassium Alum Concentration

Figure 46: Apparent viscosity as a function of potassium Alum Concentration

Figure 47: Bingham yield point as a function of potassium Alum Concentration

Filter Press Test results and Dissection:

The results of this test on the mud samples had small deviations from the base mud sample. The base mud filtrate was 6.8 mL and yielded a 0.9 mm mud cake, while the 0.5 lb/bbl potassium alum sample had 6.7 mL filtrate and 1 mm mud cake. Also, the 0.6 lb/bbl potassium alum sample yielded 6.65 mL filtrate and 1.2 mm mud cake. The 0.7 lb/bbl potassium alum sample yield 6.9 mL filtrate and 1.3 mud cake. Finally, the 1 lb/bbl potassium alum sample had 7 mL filtrate and 1.7 mm mud cake. In general, all mud samples had relatively low filtration rates and yielded thin, low permeability mud cakes. Also, it is important to point that all mud cakes from this test were within the field recommended thickness of not exceeding 2 mm. Table 20 show the filtrate volume in milliliter and mud cake thickness in millimeter results from the filter press tests different muds tested.

Mud	Filtrate mL	Mud Cake mm
Base	6.8	0.9
Base+ 0.5 lb/bbl Potassium Alum	6.7	1
Base+ 0.6 lb/bbl Potassium Alum	6.65	1.2
Base+ 0.7 lb/bbl Potassium Alum	6.9	1.3
Base+ 1 lb/bbl Potassium Alum	7	1.7

Table 20: Filter Press Test Results Summary

Figures 48 and 49 show the filter press results for muds tested with respect to Potassium Alum concentration in lb/bbl.

Figure 48: Filtrate volume at different concentrations of potassium alum

Figure 49: Filtrate volume with different concentrations of potassium alum

Figures 50 and 51 show the mud cake thickness results in millimeter from the filter press test for muds tested with respect to Potassium Alum concentration in lb/bbl.

Figure 50: Mud cake thickness at different potassium alum concentrations

Figure 51: Mud cake thickness as a function of potassium alum concentration

Resistivity Test Results:

Table 21 show the results summary for resistivity tests performed on the filtrate and mud cake yielded from the muds tested with different concentrations of Potassium Alum in lb/bbl.

Mud	Filtrate Resistivity Ω	Mud Cake Resistivity Ω
Base	0.624	0.72
Base+ 0.5 lb/bbl Potassium Alum	0.582	0.706
Base+ 0.6 lb/bbl Potassium Alum	0.63	0.85
Base+ 0.7 lb/bbl Potassium Alum	0.613	0.702
Base+ 1 lb/bbl Potassium Alum	0.575	0.673

Table 21: Filtrate & mud cake resistivities results summary at different concentration of potassium alum

Figures 52-55 show the results for resistivity tests performed on the filtrate and mud cake from the muds tested with different concentrations of Potassium Alum in lb/bbl.

Figure 52: Filtrate resistivity at different potassium alum concentrations

Figure 53: Filtrate resistivity as a function of potassium alum concentration

Figure 54: Mud cake resistivity at different potassium alum concentrations

Figure 55: Mud cake resistivity as a function of potassium alum concentration

Filtrate as Drinking water test Results:

For this test a water safety kit was used. The kit was based on the U.S. Environmental Protection Agency (EPA) guideline standards. The kit was designed to identify eight common contaminants, which are:

- 1- Bacteria
- 2- Lead
- 3- Pesticide
- 4- Nitrate
- 5- Nitrite
- 6- pH
- 7- Hardness
- 8- Chlorine

This test was not intended to prove the drinkability of the mud filtrate, rather than how safe this mud is to be used and does not impose health or environmental hazards. The test was carefully conducted on the filtrate from the 0.7 lb/bbl potassium alum mud sample. The filtrate had to sit in a special bacteria test vial for 48 hours before confirming the result. The final result indicated no bacteria was detected. The rest of the tests were carried with test strips special for each one and all passed, except for the pH and hardness tests.

The pH level of the filtrate was around 10.8, which was part of the mud design to prevent the aluminum species from precipitating out the mud. Nonetheless, given that the mud has a low filtration rate; it is most likely that the filtrate will be diluted quickly if it somehow finds its way through a water bearing formation. The filtrate hardness was actually less than 85 ppm. When

comparing the test strip to the color coded table from the test kit, it was slightly darker than the 50 ppm label. However, a conservative approach was taken by adding the next higher value (120 ppm) on the reference table and taking the middle point. The hardness of the filtrate is caused by the mineral content in it, mainly the aluminum in this mud design. However, once the filtrate is diluted and pH value fall below 10, the aluminum species will precipitate out of the water as aluminum hydroxide or aluminum minerals by reacting with ions in the water.

Contaminant	Measurement	EPA Standard level	Result
Bacteria	None	None	Passed
Lead	None	< 15 ppb	Passed
Pesticide	None	< 3 ppb	Passed
Total Nitrate/Nitrite	0	< 10 ppm	Passed
Nitrite	0	< 1 ppm	Passed
pH	10	6.5 to 8.5	Failed
Hardness	85	≤ 50ppm	Failed
Total Chlorine	0	< 4 ppm	Passed

Table 22: Water safety kit results summary

Conclusion

The aluminum chemistry proved to be very effective to control swelling of the Marcellus shale. The 0.7 lb/bbl potassium alum mud design completely suppress the swelling of shale. Also, the choice of using potassium alum as the aluminum hydroxide source was very successful. It had double inhibition actions; precipitating chemistry and ion exchange as it provided potassium ions along with the aluminum. This eliminated the need to add potassium chloride KCl to enhance the performance of the mud, thus eliminating the chlorine byproduct. The shale swelling was reduced from approximately 5 % to zero percent with the use of the 0.7 lb/bbl potassium alum in the mud.

Also, all mud components used were naturally occurring and biodegradable. The double inhibitive action of the additive resulted in using a low concentration. This added to the mud's handling/environmental safety and its economical feasibility. Although the mud is not to be used for human consumption, the drinking water safety kit proved that the mud filtrate has no major impact as a water contaminant. The pH and hardness could be easily treated. However, drilling fluid disposal should comply with local, state, and federal regulations.

Finally, I recommend putting the mud through an aging cell to test how well the mud holds its rheological properties. Also, I recommend performing swelling test at elevated temperature and pressure levels to examine their effect on the inhibition quality of the mud at near down-hole conditions.

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Appendix

Swelling Experiments Log

1- Water Baseline:

Sample #	21
Initial Weight (g)	26.6
Final Weight (g)	26.3
Initial Height (mm)	3.427
Swelling Height (mm)	0.169
Swelling %	4.93
Remarks	Tested with dynamic swelling meter

Sample #	17
Initial Weight (g)	25.6
Final Weight (g)	25.4
Initial Height (mm)	3.302
Swelling Height (mm)	0.161
Swelling %	4.88
Remarks	Tested on dial indicator

Sample #	19
Initial Weight (g)	23.8
Final Weight (g)	23.7
Initial Height (mm)	3.074
Swelling Height (mm)	0.156
Swelling %	5.07
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
17	3.302	0.161	4.88
19	3.074	0.156	5.07
21	3.427	0.169	4.93
Average			4.96

2- Base Mud:

Sample #	14
Initial Weight (g)	32.3
Final Weight (g)	32.0
Initial Height (mm)	4.162
Swelling Height (mm)	0.115
Swelling %	2.76
Remarks	Tested with dynamic swelling meter

Sample #	18
Initial Weight (g)	31.7
Final Weight (g)	31.5
Initial Height (mm)	4.084
Swelling Height (mm)	0.120
Swelling %	2.94
Remarks	Tested on dial indicator

Sample #	12
Initial Weight (g)	28.0
Final Weight (g)	27.7
Initial Height (mm)	3.607
Swelling Height (mm)	0.109
Swelling %	3.02
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
18	4.084	0.120	2.94
12	3.607	0.109	3.02
14	4.162	0.115	2.75
Average			2.90

3- 1 lb/bbl Potassium Alum Concentration :

Sample #	28
Initial Weight (g)	47.5
Final Weight (g)	47.1
Initial Height (mm)	6.107
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested with dynamic swelling meter

Sample #	23
Initial Weight (g)	44.0
Final Weight (g)	43.6
Initial Height (mm)	5.660
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

Sample #	11
Initial Weight (g)	44.0
Final Weight (g)	43.6
Initial Height (mm)	7.128
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
28	6.107	0	0
23	5.660	0	0
11	7.128	0	0
Average			0

4- 0.5 lb/bbl Potassium Alum Concentration:

Sample #	16
Initial Weight (g)	45.2
Final Weight (g)	44.7
Initial Height (mm)	6.652
Swelling Height (mm)	0.093
Swelling %	1.40
Remarks	Tested with dynamic swelling meter

Sample #	15
Initial Weight (g)	43.3
Final Weight (g)	42.9
Initial Height (mm)	5.574
Swelling Height (mm)	0.079
Swelling %	1.42
Remarks	Tested on dial indicator

Sample #	13
Initial Weight (g)	49.6
Final Weight (g)	49.4
Initial Height (mm)	6.990
Swelling Height (mm)	0.098
Swelling %	1.40
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
15	5.574	0.079	1.42
13	6.99	0.098	1.40
16	6.652	0.093	1.40
Average			1.41

5- 0.6 lb/bbl Potassium Alum Concentration:

Sample #	8
Initial Weight (g)	36.6
Final Weight (g)	36.3
Initial Height (mm)	5.764
Swelling Height (mm)	0.072
Swelling %	1.25
Remarks	Tested with dynamic swelling meter

Sample #	5
Initial Weight (g)	36.4
Final Weight (g)	36.1
Initial Height (mm)	5.190
Swelling Height (mm)	0.064
Swelling %	1.23
Remarks	Tested on dial indicator

Sample #	3
Initial Weight (g)	34.1
Final Weight (g)	33.7
Initial Height (mm)	5.619
Swelling Height (mm)	0.070
Swelling %	1.25
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
3	5.619	-----	-----
5	5.190	0.064	1.23
8	5.764	0.072	1.25
Average			1.24

6- 0.7 lb/bbl Potassium Alum Concentration:

Sample #	10
Initial Weight (g)	31.5
Final Weight (g)	31.2
Initial Height (mm)	5.024
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested with dynamic swelling meter

Sample #	2
Initial Weight (g)	31.1
Final Weight (g)	30.7
Initial Height (mm)	4.815
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

Sample #	6
Initial Weight (g)	32.3
Final Weight (g)	31.9
Initial Height (mm)	4.307
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

7- 0.7 lb/bbl Potassium Alum Concentration: (Redo/ Confirmation)

Sample #	7
Initial Weight (g)	47.1
Final Weight (g)	46.7
Initial Height (mm)	7.005
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested with dynamic swelling meter

Sample #	5
Initial Weight (g)	27.7
Final Weight (g)	27.4
Initial Height (mm)	4.930
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

Sample #	1
Initial Weight (g)	26.5
Final Weight (g)	26.2
Initial Height (mm)	3.705
Swelling Height (mm)	0
Swelling %	0
Remarks	Tested on dial indicator

Results Summary:

Sample #	H Initial (mm)	Swelling Height(mm)	Swelling %
2	4.815	0	0.00
6	4.307	0	0.00
10	5.024	0	0.00
5	4.930	0	0.00
1	3.705	0.001	0.03
7	7.005	0	0.00
Average			0.01